Advanced Ceramic Materials and Packaging Technologies for Realizing Sensors for Concentrating Solar Power Systems

Sporian Microsystems, Inc.
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Subcontractor:
University of Wisconsin Thermal Hydraulic Laboratory

Project start date: November 15, 2012

Outline

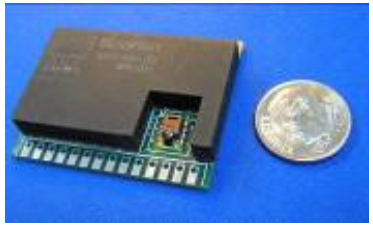
- About Sporian Microsystems, Inc.
- Review Sporian's HT Sensor Technology
- Project Motivation and Background
- Phase I Objectives, Innovation and Approaches
- Technical Results and Analysis
- Significance of the Results and Challenges
- Project Milestones to Date
- Accomplishments and Future Work Planned

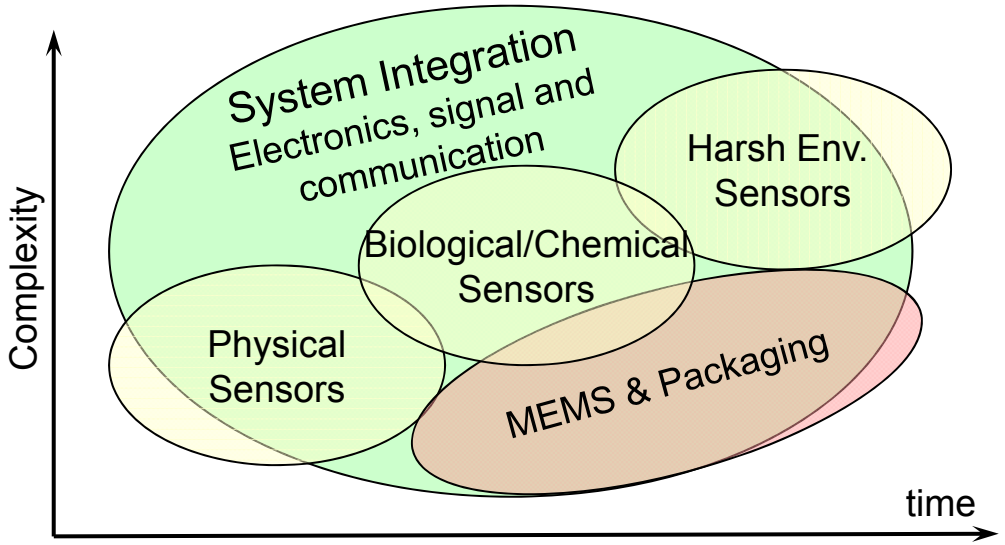
SPORIAN MICROSYSTEMS, INC

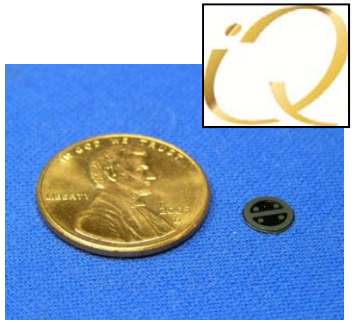
Sporian Overview and Technology Focus

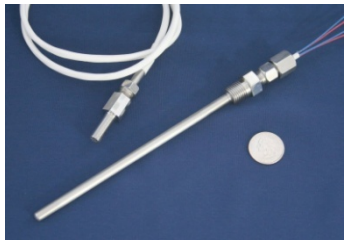
- **Founded in 2000, focuses on advanced sensors, packaging and systems**
- **Develop and commercialize sensors/systems for a range of industries:**
  - Energy Generation ● Aerospace and Transportation
  - Environmental Safety ● Water Health Management ● Biomedical
  - Asset Monitoring ● Integrated Vehicle Health Monitoring ● Homeland Security

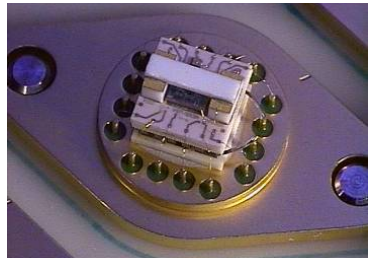
best of sensors expo2008
Bronze Level Winner

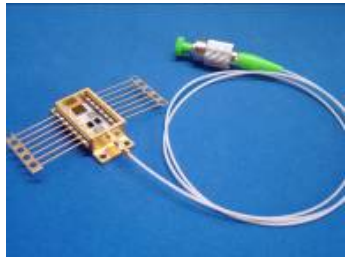












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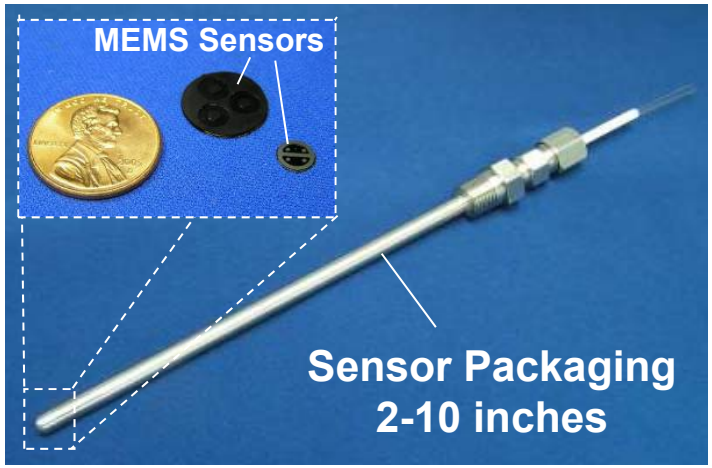
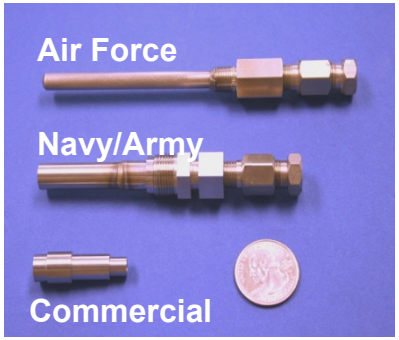
Sporian High-temperature Harsh Environment MEMS Sensors and Packaging

R&D Efforts for High-temperature Sensors and Packaging:

- Directly monitor the most harsh environments and costly components
  - High-temperature: up to **1400°C**
  - High-pressure: up to **1000 psi**
  - ▪ Temperature ▪ Pressure ▪ Flow sensors
  - **Energy Generation Applications**
    - Concentrated Solar Power (CSP)
    - Nuclear Power Generation
    - Fossil Fuel (Gas/Coal) Turbine Applications
  - **Aerospace Applications**
    - Aerospace conformal sensor packaging
    - Smart sensor system





- Packaging is critical to facilitate sensor utility in various environments

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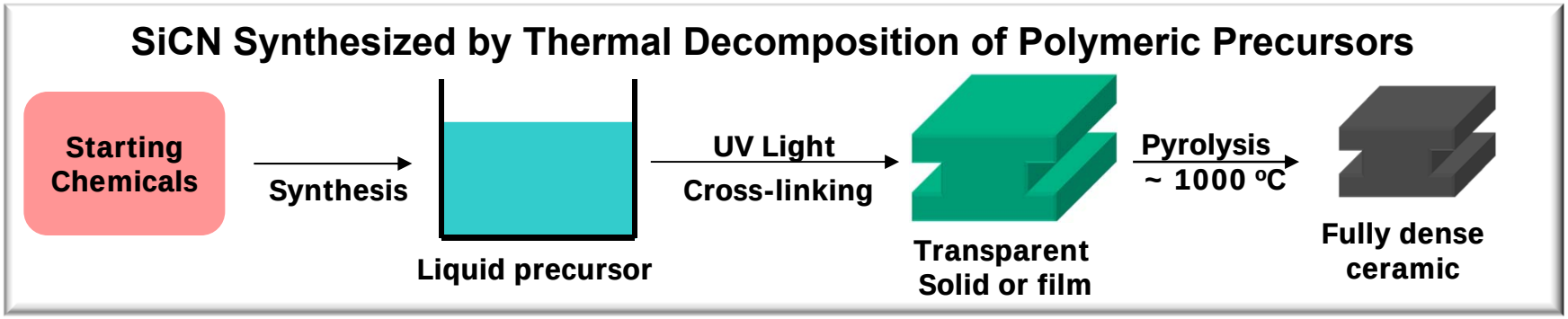
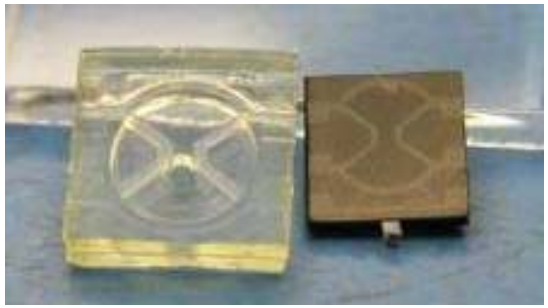
Sporian SiCN Sensor Technology

Core Technology:

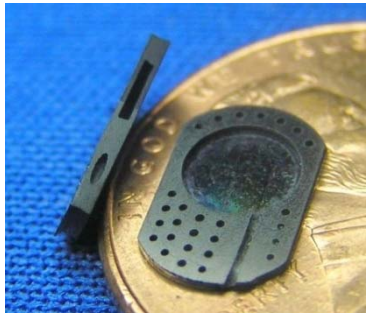
- **Proprietary Polymer Derived SiCN Materials and Micro-fabrication Process**





Polymer Form and SiCN

Batch Fabrication



Complex Features

Prior Demonstrated HT SiCN Sensor Technology:

- Thermo/piezo-resistive temperature/pressure sensor suite
- Capacitive based pressure sensor
- Hot wire based flow sensors

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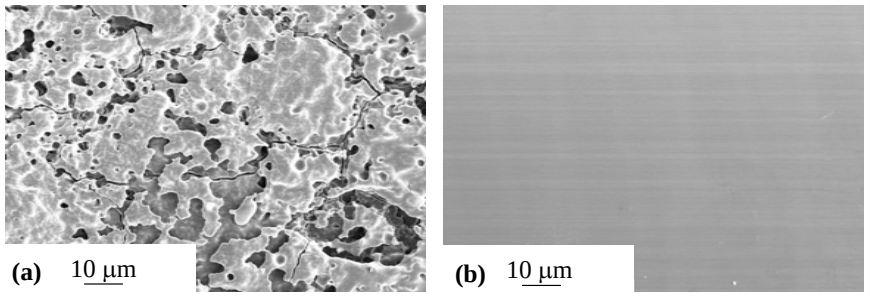
Key Benefits of SiCN

- **Excellent High-temperature Thermo-mechanical Properties**
  - Thermally stable and resists large scale crystallization up to 1800 ^{o}C
  - Creep resistance exceeds state-of-the-art polycrystalline SiC and Si_3N_4
  - High thermal shock resistance due to absence of grain boundary phases

- **Excellent High-temperature Oxidation/Corrosion Resistance**
  - Demonstrated material strength and resistance over SiC
  - Oxidation/corrosion resistance improved significantly by doping

After 1100^{o}C - 400 hr in 100% H_2O Vapor



un-doped SiCN Al-doped SiCN

- **Tunable Electrical Properties**
  - Conductivity, piezo-resistivity and dielectric properties
- **Facilitates Micro-fabrication**
  - Multi-layer, multi-formulation photo-lithography, laser-cutting, molding etc.

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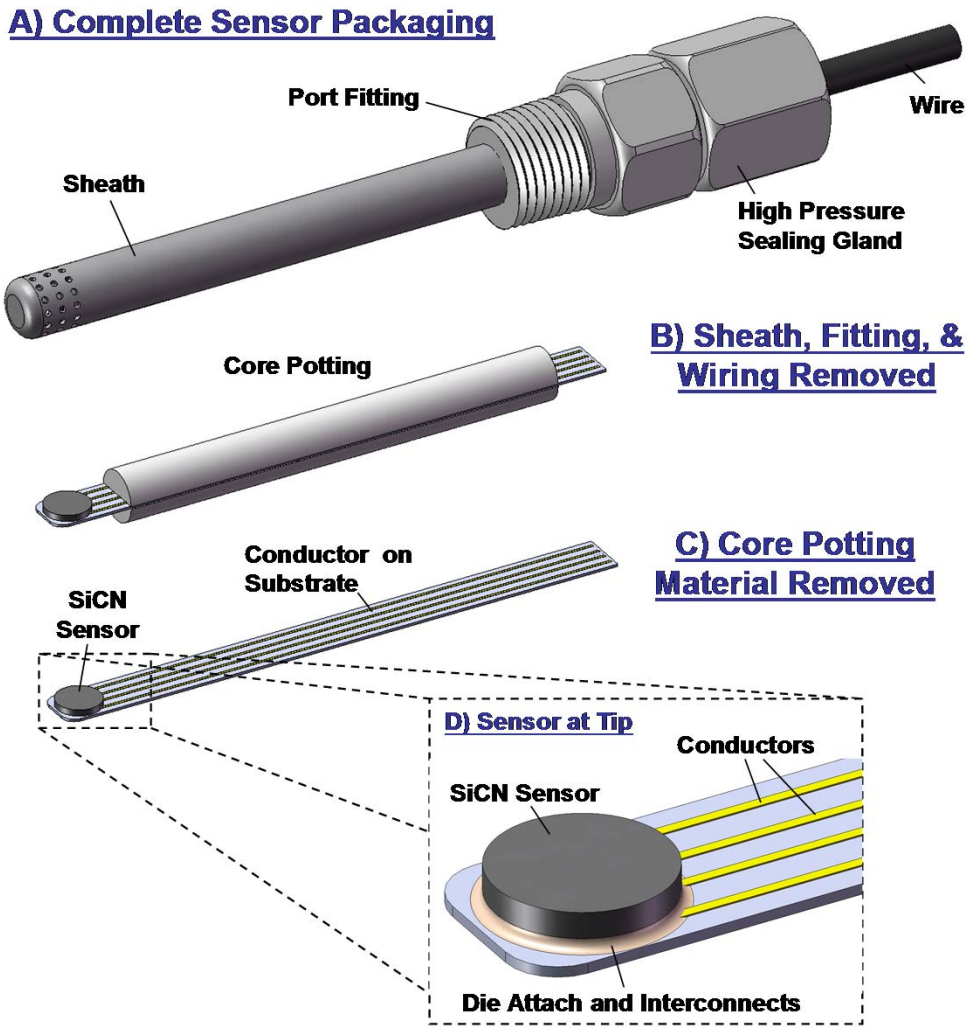
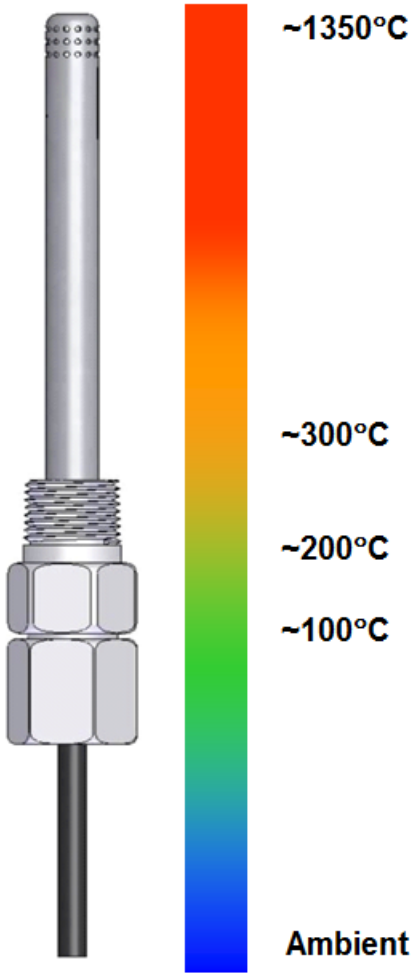
Sporian Sensor Packaging Technology

High-temperature Sensor Packaging:

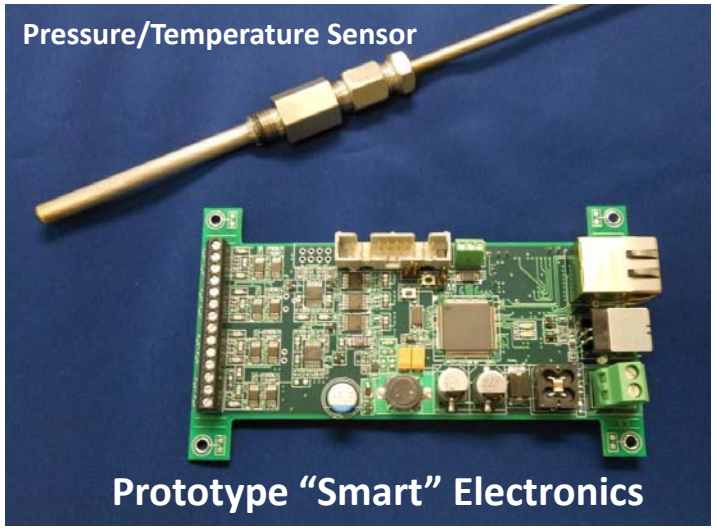


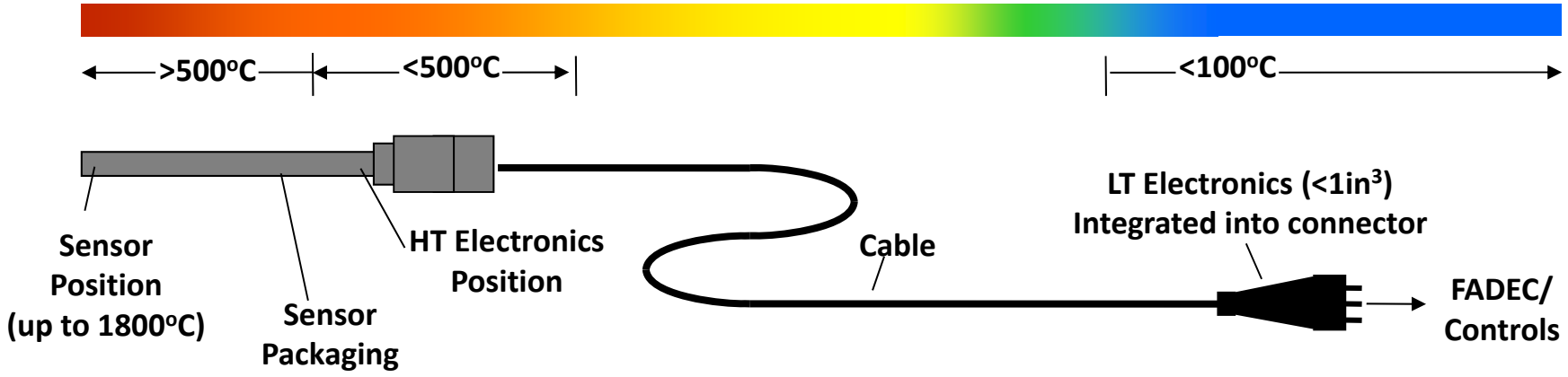


Future Electronic Packaging

Strong Pull from OEMs to Add "Smart" Functionality to Sporian Existing Sensor and Packaging Technology

- Digital/bus interface
- Internal compensation/calibration
- Internal health check/indication
- Implemented with HT electronics
- Small electronics: Bump on cable





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Project Motivation and Background

- **Needs of Concentrated Solar Power Systems:**
  - Robust sensing systems for safety/efficiency monitoring and control
- **Primary Technical Challenges:**
  - Extremely harsh working conditions
    - **High Operating Temperature (HOT) fluids**
    - **Highly corrosive molten salt environments**
- **Preliminary Solar Salt Testing of Sporian SiCN at Sandia National Laboratory:**
  - **SiCN tested in nitrate(60/40) solar salt at 300°C for 500 hr**
  - No visible surface corrosion or measurable mass change
  - Demonstrated SiCN as a potential high-T sensing material in HOT fluid environments for CSP applications
- **Long-Term Goal:**
  - Leverage Sporian sensor/packaging technology to support applications in CSP systems:

▪ Pressure ▪ Flow ▪ Temperature ▪ Level sensors

Phase I Objectives and Approaches

1. **Experimentally evaluate the suitability of SiCNs as *innovative* sensing materials in CSP HOT fluids**
2. **Based on the results, develop *innovative* sensor and packaging concepts for future development**

Participant Roles and Approaches:

- **Sporian:**
  - SiCN sensor material identification and sample fabrication
  - Pre- and post-test material testing and evaluation
  - Conceptual sensor/packaging designs and preliminary prototyping
- **Subcontractor, Consultant and in-kind Support:**
  - **Thermal Hydraulic Laboratory at the University of Wisconsin (UW)**
    - Consult on CSP sensor environments and operation parameters
    - Identify potential molten salts and conduct HOT fluid testing
    - Pre- and post-test corrosion-resistivity evaluation

  - **CSP subject matter experts and establish system/sensor OEMs**
    - Consult on sensor/package requirements and specification development

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Key Technical Requirements

Identification of Overall Operational and Interface Requirements

- Operation environments: Daily T-cycling and system draining
- Challenges: HT molten salt corrosion and flow erosion
- Flow spinning, turbulence and vibration caused damages
- Extreme scenarios: System solidification and re-melting
- Identified 4 potential sensor types of high interest: T/P, flow, level
- Identified sensor locations and measurement ranges/resolutions
- Identified candidate packaging materials: Ni-superalloys, etc.
- Established Key requirements for sensor and packaging design
- Identified critical factors for hardware implementation
- Identified follow-on system integration standards and approaches

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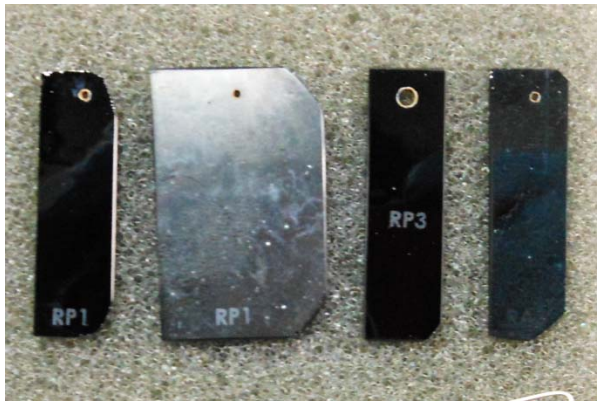
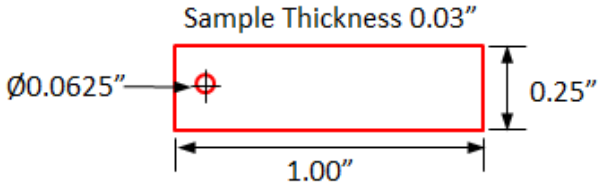
Preparation of SiCN Samples

Identification of SiCN Formulations for Phase I Evaluation Efforts

- High temperature material stability and electrical conductivity
- Excellent oxidation and corrosion resistance for CSP applications
- Characterization of the material/mechanical/electrical properties
- Design and fabricate SiCN coupons for HOT fluid testing in UW



SiCN Coupons for Molten Salt Testing



Four Groups of SiCN Samples Measured before Testing

SiCN Sample Weight (gram)	Salt-1 (S1) Nitrate	Salt-2 (S2) Carbonate	Salt-3 (S3) Chloride	Reference (R)
P1	0.2677	0.2742	0.2520	0.2507
A1	0.6133	0.5741	0.6051	0.5936
P3	0.2407	0.2299	0.2176	0.2698
A3	0.2818	0.2966	0.2991	0.2833

Nickel Wire was Used to Hold the Samples

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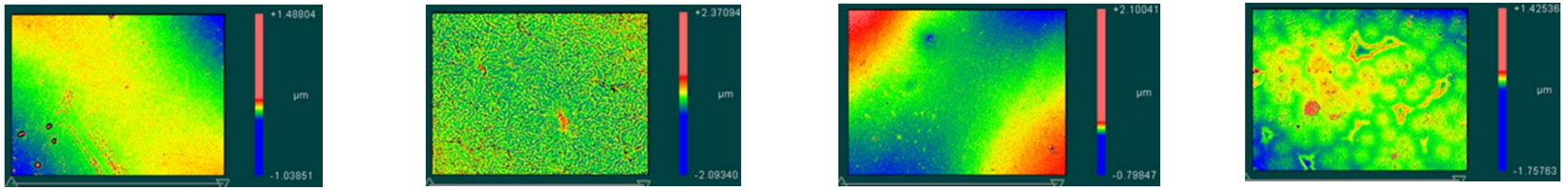
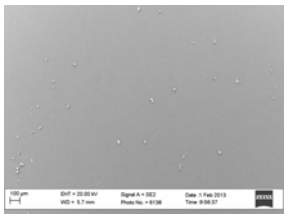
Technical Analysis Approaches

- **Weight and Dimension Measurements and Analysis**
- **Surface Characteristics and Analysis: Microscope, Profilometer and SEM**
- **Elemental Analysis and Chemical Characterization: EDS**
- **Mechanical Strength and Electrical Properties Evaluation** (as appropriate)

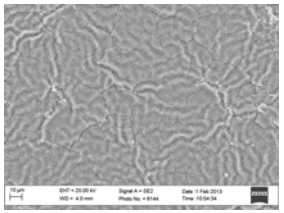
Surface Roughness Maps of SiCN Reference Samples



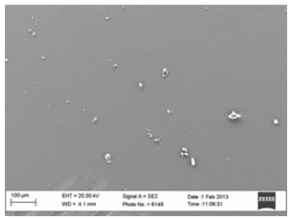
SEM Surface Scans of SiCN Reference Samples



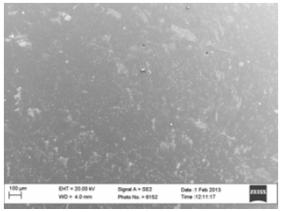
P1: Low magnification show very little structure.



P1W: High mag. shows structure and surface cracks

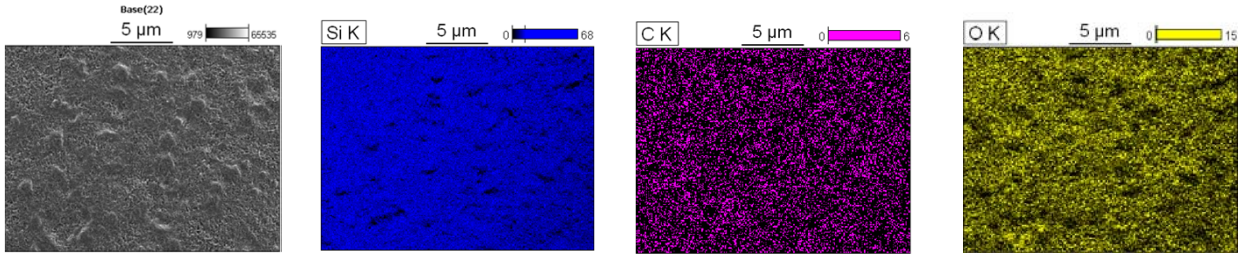


P3: Low magnification show very little structure



A3: Low mag. shows pits and some surface marks

EDS Elemental Analysis of Reference Sample:



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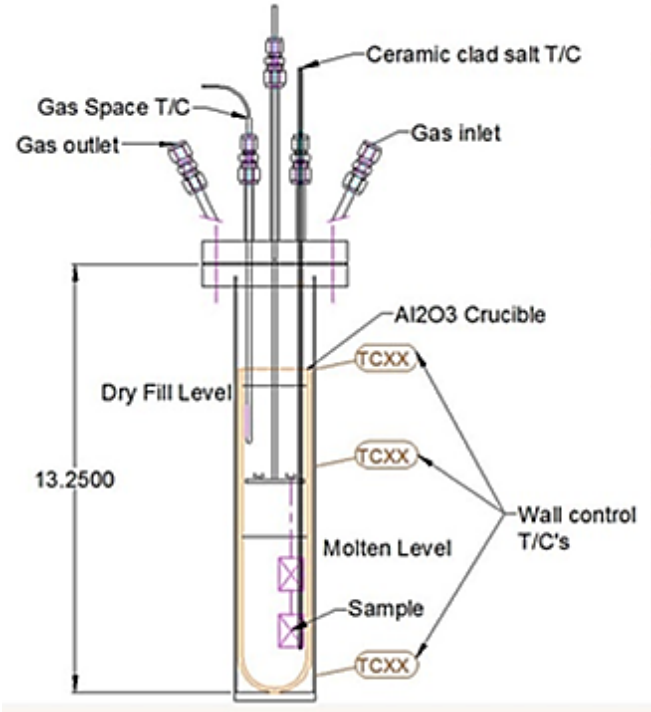
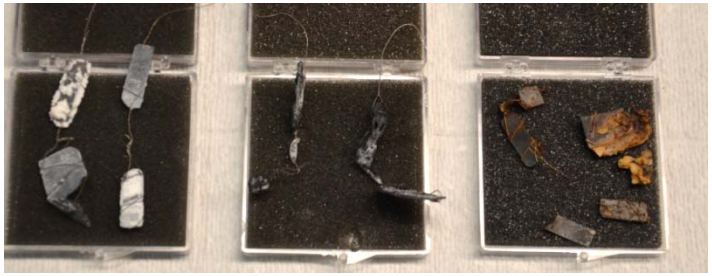
500hr Solar Salt Exposure Testing

3 Different CSP Relevant Inorganic Salts and Test Facility in UW

HOT Fluid Mixture	M.P. [°C]	T.S.B.P. [°C]	Test T [°C]	Duration [hour]	Cp [J/g-K]	V.P. @800°C	Corrosion with SS	Cost [$/kg]
DOE's Target	250	800			1.5	<1atm	Excellent	<1
$NaNO_3$ KNO_3	228	600	**550°C**	**500 hr**	1.5	<1atm	Good	~1
$K_2LiNa_2CO_3$	397	>830	**650°C**	**500 hr**	1.8	<1atm	Fair	~2
KCl $MgCl_2$	426	>1418	**750°C**	**500 hr**	na	<0.1atm	Fair	~0.2



SiCN Samples before/after Cleaning



Nitrate Salt 550 °C **Carbonate 650 °C** **Chloride Salt 750 °C**

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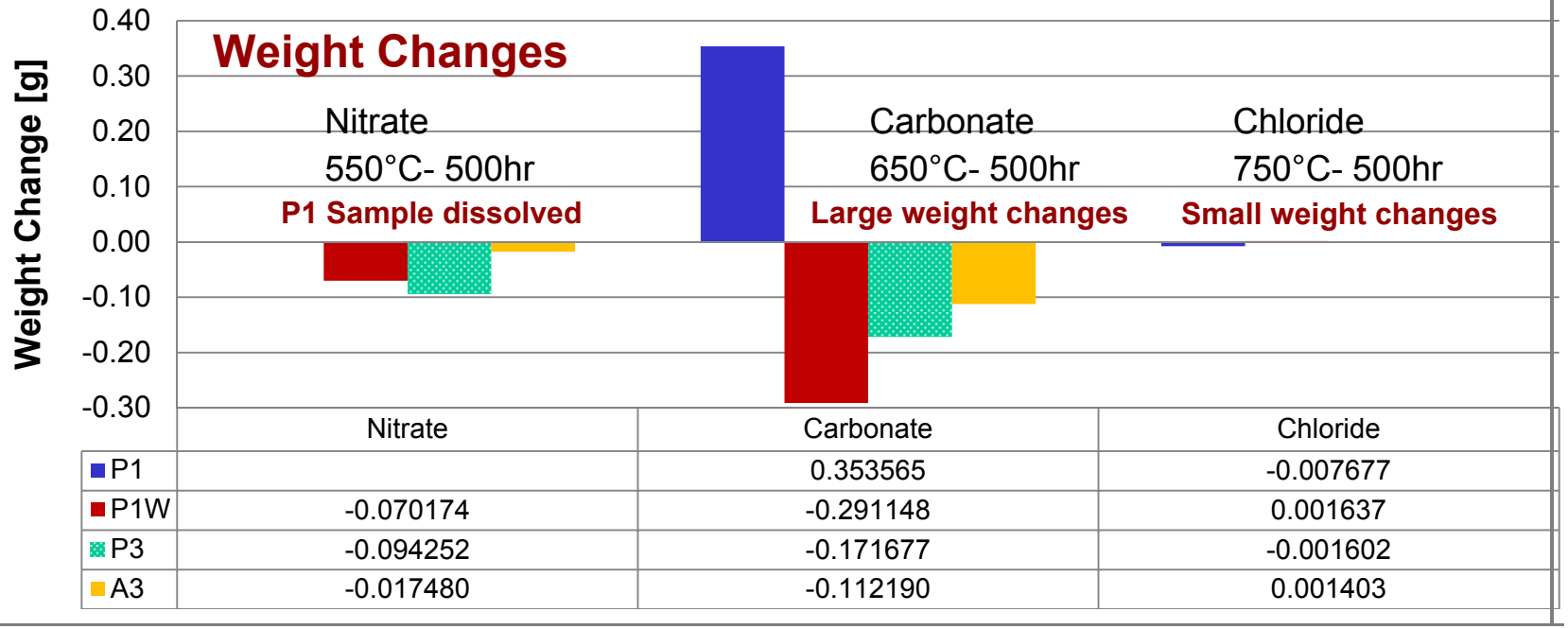
Key Technical Results and Analysis



	Nitrate	Carbonate	Chloride
P1		0.353565	-0.007677
P1W	-0.070174	-0.291148	0.001637
P3	-0.094252	-0.171677	-0.001602
A3	-0.017480	-0.112190	0.001403

SiCN Sample Specification and Weight Loss (%)

Laser Mark	Formulation	Heat-treatment	Nitrate	Carbonate	Chloride
P1	#1	Pyrolized	N/A	129%	-3.0%
P1w (A1)	#1	Annealed	-11%	-51%	0.3%
P3	#3	Pyrolized	-39%	-75%	-0.7%
A3	#3	Annealed	-6%	-38%	0.5%

Remarks:

1. SiCN formulation-3 showed better corrosion resistance than formulation-1
2. Annealed samples showed less weight loss than pyrolyzed counterparts

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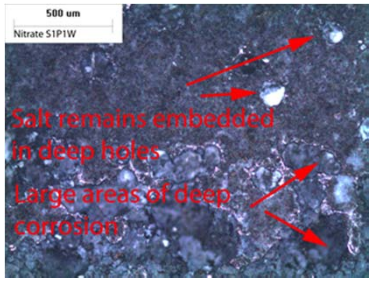
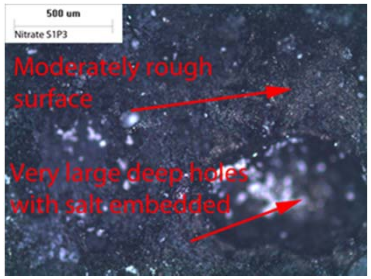
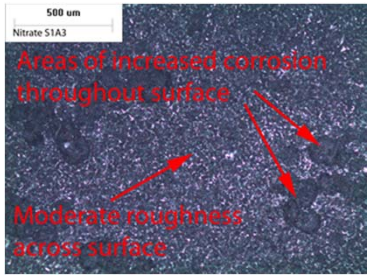
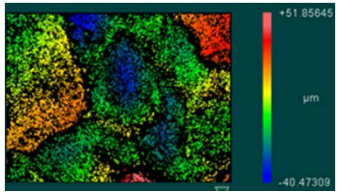
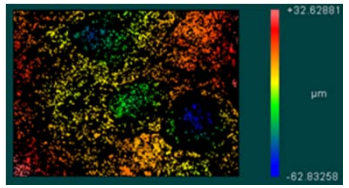
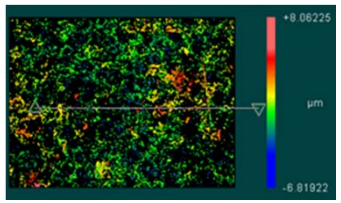
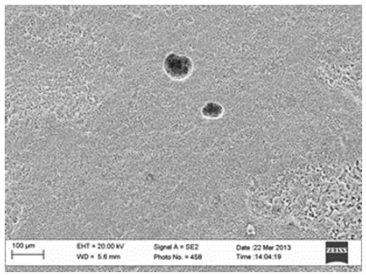
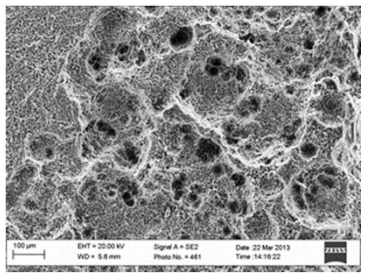
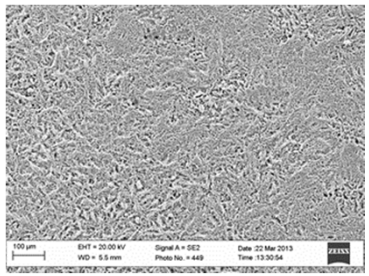
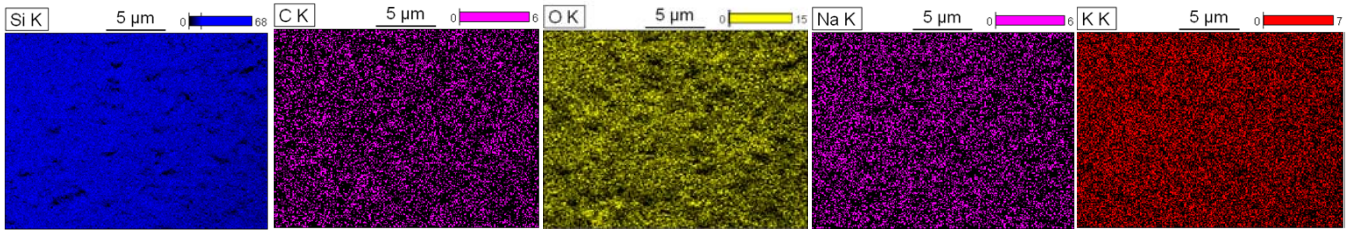
Nitrate Salt Test Results and Analysis

550°C-500hr

	S1P1	S1P1W	S1P3	S1A3
Microscope Images: Moderate corrosion and pits/holes	N/A Post-test sample dissolved upon cleaning			
Surface Roughness: Increased from ±2µm to ±50µm				
SEM Images: Moderate corrosion and small to large pits				

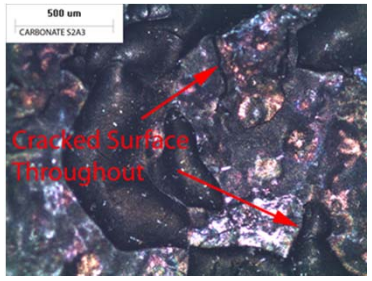
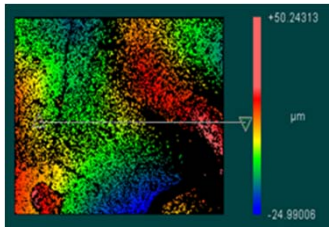
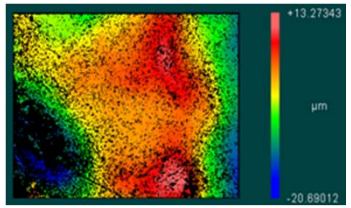
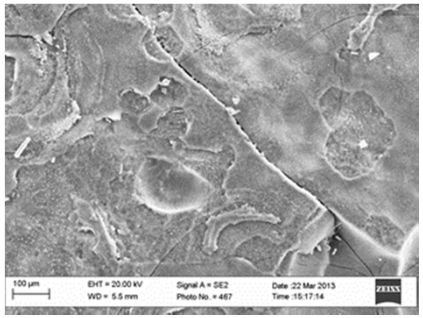
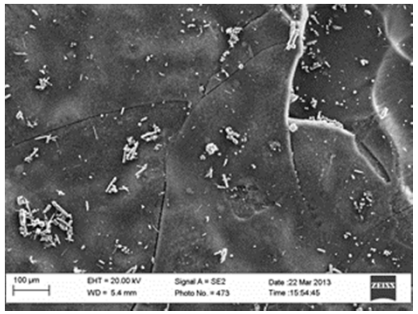
EDS Elemental Analysis:

No distinct elemental characteristics, suggests dissolving other than compound formation



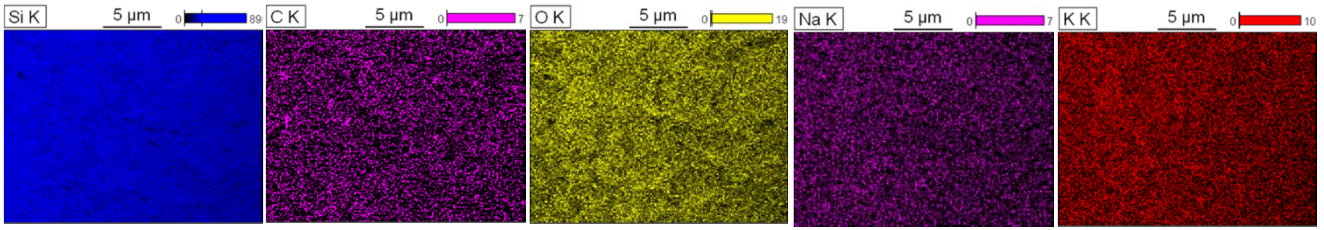
Carbonate Salt Test Results and Analysis

650°C-500hr

	S2P1	S2P1W	S2P3	S2A3
Microscope Images: Severe corrosion and damages	N/A Post-test sample crumbled		N/A Post-test sample crumbled	
Surface Roughness: Increased from ±2µm to ±50µm				
SEM Images: Severe corrosion, pitting, crack, swelling and delamination				

EDS Elemental Analysis:

No distinct elemental characteristics on sample surface



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Chloride Salt Test Results and Analysis

750°C-500hr

Microscope Images:

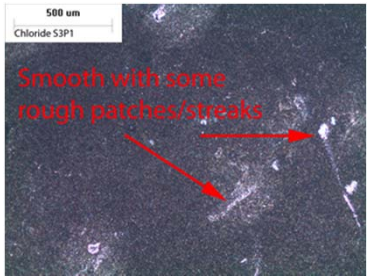
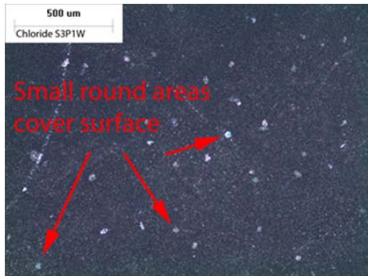
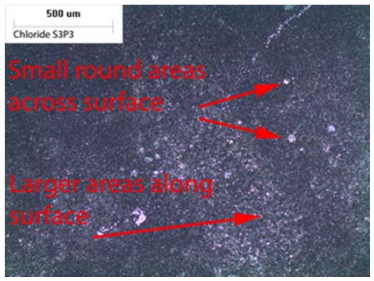
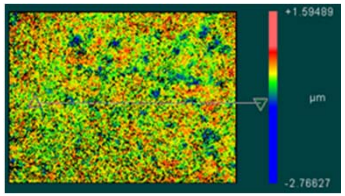
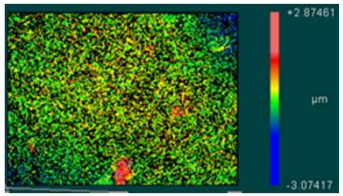
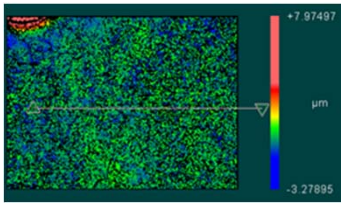
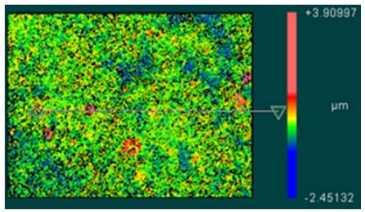
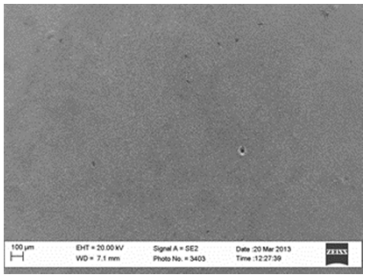
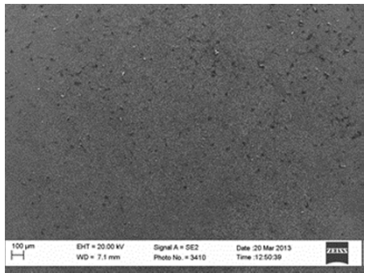
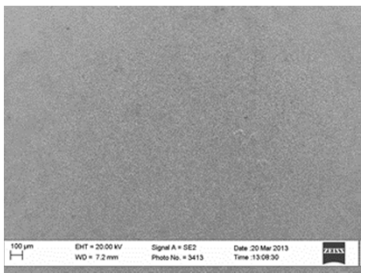
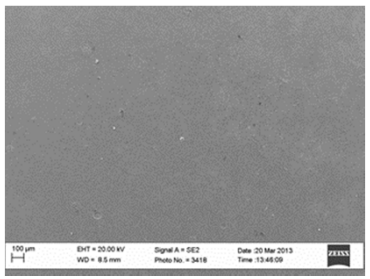
Minor corrosion; rough patches

Surface Roughness:

Small increases from ±2 to ±5µm

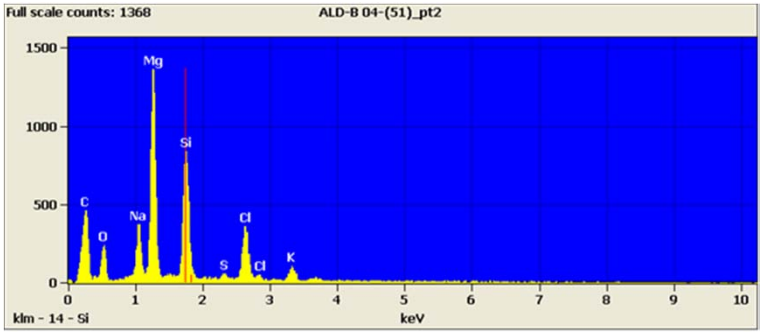
SEM Images:

Minor corrosion; smooth surfaces

S3P1	S3P1W	S3P3	S3A3
			
			
			

EDS Point Scan Analysis:

- Point scans show similar results as full EDS maps.
- No distinct characteristics between different locations.
- No oxide or other compound formations.



SPORIAN MICROSYSTEMS, INC

Significance of the Results and Challenges

Significance of the Results:

- SiCN materials are thermally and chemically stable in high-T molten salts.
- Corrosion mechanism is mainly dissolving started at surface defects.
- SiCN samples showed good corrosion resistance in 750°C-500hr chloride test.
- Some of the SiCNs showed better corrosion resistance trend than the others.
- Protective packaging is needed to avoid molten salt attack in nitrate and carbonate.

Challenges:

- **Corrosive molten salt environments for direct-contact sensors**
- **Need protective packaging design for pressure and flow sensors**

Important Milestones:

- End of Month 4: A fundamental understanding/definition of key requirements for practical implementation of proposed hardware technology ***(completed).***
- End of Month 4: Identified optimal SiCN formulations for CSP applications and fabricated test coupons for Phase I corrosion evaluations ***(completed).***
- End of Month 6: Evaluation and feasibility demonstration of choice SiCN ceramics in relevant HOT fluids for CSP applications ***(completed).***
- End of Month 9: Preliminary designs and prototyping of the sensors, packaging and electronics, and a definitive development plan for Phase II ***(currently ongoing).***

SPORIAN MICROSYSTEMS, INC

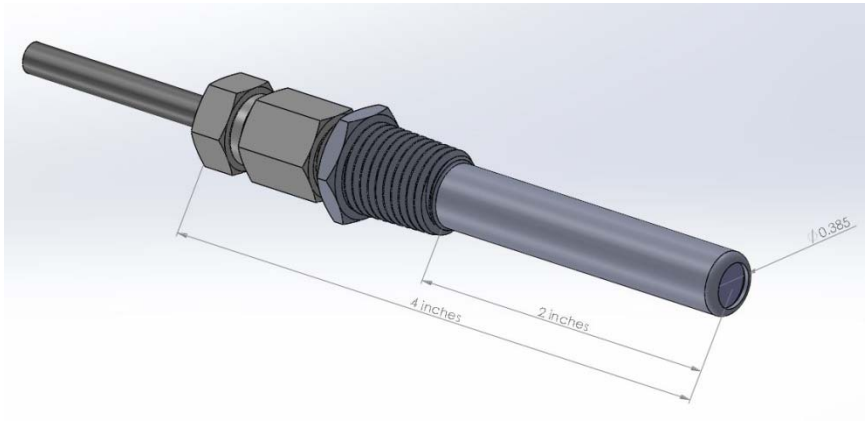
Innovated CSP Sensors and Packaging

Primary Objective:

- Design concepts for sensor, packaging, and electronics.
- Sensor types: temperature, pressure, flow and level sensors

Accomplishments/Progress Update:

- Preliminary design of pressure sensor and packaging.
- Investigation on suitable flow and level sensor design concepts.



Future Work Planned:

- Identify materials, designs and technologies to be leveraged.
- Evaluation matrix for sensor design and packaging configurations.
- Analytical and computational modeling of performance.
- Define preliminary hardware designs and assembly processes.
- Concepts for near and long-term development (Phase II and beyond).
- Potential Phase II: Define short/long term testing and implementation strategies.
- Potential Phase II: Electronic and CSP control/monitoring system interface .